PB86-156486

Design and Construction of a Mobile Activated Carbon Regenerator System

MSA Research Corp., Evans City, PA

Prepared for

Environmental Protection Agency, Cincinnati, OH

Jan 86

U.S. Department of Commerce
National Technical Information Service
NTIS

PB86-156486

EPA/600/2-86/015
January 1986

DESIGN AND CONSTRUCTION OF A MOBILE ACTIVATED CARBON REGENERATOR SYSTEM

by

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Contract No. 68-03-2110

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CINCINNATI, OHIO 45268

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U.S. DEPARTMENT OF COMMERCE
SPRINGFIELD, VA. 22161

TECHNICAL REPORT DATA
(Please read Instructions on the reverse before completing)

1. REPORT NO.	2.	3. RECIPIENT'S ACCESSION NO.
EPA/600/2-86/015		PB86 156486/AS

4. TITLE AND SUBTITLE	5. REPORT DATE
DESIGN AND CONSTRUCTION OF A MOBILE ACTIVATED CARBON REGENERATOR SYSTEM	January 1986
	6. PERFORMING ORGANIZATION CODE: A-918

7. AUTHOR(S)	8. PERFORMING ORGANIZATION REPORT NO.
R.H. Hiltz	MSAR 81-107

9. PERFORMING ORGANIZATION NAME AND ADDRESS	10. PROGRAM ELEMENT NO.
MSA Research Corporation Division of Mine Safety Appliances Co. Evans City, Pennsylvania 16033	11. CONTRACT/GRANT NO. 68-03-2110

12. SPONSORING AGENCY NAME AND ADDRESS	13. TYPE OF REPORT AND PERIOD COVERED
Hazardous Waste Engineering Research Laboratory Office of Research and Development U.S. Environmental Protection Agency Cincinnati, Ohio 45268	Final-8/6/74-2/8/79 14. SPONSORING AGENCY CODE: EPA/600/12

15. SUPPLEMENTARY NOTES

Project Officer: John E. Brugger (201)321-6634

16. ABSTRACT

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This program was undertaken to assess the feasibility of designing and building a mobile carbon regeneration unit - including an incinerator/scrubber to destroy the offgases - for field use in conjunction with mobile carbon adsorption systems. A system was designed and built based on technology developed in the earlier fabrication of a laboratory-sized regenerator and on an in-depth evaluation of factors affecting system design and size. Housed in a standard van-type of trailer, the system met all weight and size limitations for over-the-road transportation. The system includes a direct fired, rotating barrel kiln to thermally regenerate the carbon, an incinerator and scrubber to destroy the desorbed materials and treat the offgases, and a separator to reclaim the reactivated carbon granules.

Test runs using spent carbon from an on-site treatment of a spill were quite successful. The carbon was returned to essentially 100% activity with an 88% volume recovery. The unit has been delivered to the US EPA for their use.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group

18. DISTRIBUTION STATEMENT	19. SECURITY CLASS *(This Report)*	21. NO. OF PAGES
RELEASE TO PUBLIC	UNCLASSIFIED	54
	20. SECURITY CLASS *(This page)* UNCLASSIFIED	22. PRICE $11.95

EPA Form 2220-1 (Rev. 4-77) PREVIOUS EDITION IS OBSOLETE

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FORWARD

Today's rapidly developing and changing technologies and industrial products and practices frequently carry with them the increased generation of solid and hazardous wastes. These materials, if improperly dealt with, can threaten both public health and the environment. Abandoned waste sites and accidental releases of toxic and hazardous substances to the environment also have important environmental and public health implications. The Hazardous Waste Engineering Research Laboratory assists in providing an authoritative and defensible engineering basis for assessing and solving these problems. Its products support the policies, programs, and regulations of the Environmental Protection Agency, the permitting and other responsiblities of State and local governments and the needs of both large and small businesses in handling their wastes responsibly and economically.

Activated carbon is proving to be an invaluable tool in the cleanup of hazardous chemical storage sites and in the response to industrial and transportation disasters. The addition of a mobile regeneration system such as designed and constructed in this project will further facilitate the efforts of field workers and allow onsite destruction of the pollutants from the environment.

David G. Stephan, Director
Hazardous Waste Engineering
Research Laboratory

ABSTRACT

Activated carbon adsorption has become a standard procedure for the cleanup of contaminated water streams. To facilitate such cleanup at hazardous waste and spill sites, mobile carbon adsorption units have been constructed and are now in use. Their primary drawback is the logistics associated with the disposal of spent (contaminated) carbon and its replenishment with fresh, active carbon.

This program was undertaken to assess the feasibility of designing and building a mobile carbon regeneration unit - including an incinerator/scrubber to destroy the offgases - for field use in conjunction with mobile carbon adsorption systems. A system was designed and built based on technology developed in the earlier fabrication of a laboratory-sized regenerator and on an in-depth evaluation of factors affecting system design and size. Housed in a standard van-type of trailer, the system meets all weight and size limitations for over-the-road transportion. The system includes a direct fired, rotating barrel kiln to thermally regenerate the carbon, an incinerator and scrubber to destroy the desorbed materials and treat the offgases, and a separator to reclaim the reactivated carbon granules.

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This report was submitted in fulfillment of Contract No. 68-03-2110 by MSA Research Corporation under the sponsorship of the U.S. Environmental Protection Agency. This report covers a period from August 6, 1974 to February 8, 1979, and work was completed as of October 15, 1981.

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SECTION 1

INTRODUCTION

One of the principal techniques now in wide use to decontaminate water is granular activated carbon, which has the ability to adsorb selected classes of chemicals preferentially from aqueous solutions or dispersions. Activated carbon is characterized by a highly developed network of small pores and interstices which contribute to its high surface-to-volume ratio and, consequently, to its high adsorptive capacity.

In spill situations where hazardous chemicals have entered the water ecosystem, where materials are leaching from landfills or waste storage sites, and in other similar situations, activated carbon treatment becomes a standard practice. To facilitate the cleanup at such sites, portable carbon adsorption systems have been developed in recent years. These are trailer or skid mounted units which can be moved over the road or by air to the place where the cleanup is to be conducted.

These portable systems have proven quite versatile and effective. However, they have a significant drawback—the logistic and administrative barriers associated with the disposal of spent (contaminated) carbon and its replacement. Large quantities of carbon are usually required for cleanup operations. And though the carbon can technically be regenerated commercially it must be moved to a facility having the necessary processing equipment, and appropriate permits for carbon reactivation. Also, fresh carbon must be moved to the field site to keep the cleanup operation active. This approach to regeneration can be complicated if hazardous materials are involved (such as PCB's or TCDD) for which there are strict regulations or prohibitions affecting commercial reactivation. Commercial regeneration facility operators are unwilling to operate their equipment routinely at the temperatures needed to destroy PCB's and TCDD because of economic reasons. Further, the regeneration market for carbon contaminated with these substances is not large enough to justify the facility operator's incurring the public concern that would result if the facility were to accept such substances. Because of these problems with commercial reactivation, carbon used to treat PCB's or TCDD is currently being disposed of in chemical landfills rather than being regenerated. Thus, the toxics accumulated on the carbon are not destroyed, only stored, possibly to enter the environment in the future.

Clearly, the utility of portable activated carbon systems (particularly when used on toxic chemical contaminated carbon not commercially regenerable) could be enhanced if regeneration of the carbon could be achieved at the cleanup site. Since the adsorption system has been adapted to a mobile base, it was not difficult to conceive of a regeneration process that could be fitted onto a skid or mobile trailer bed.

Most materials that can be adsorbed by carbon can be stripped by means of steam or other thermal treatments to regenerate the adsorptive capacity of the carbon. The residue from this stripping then contains the adsorbed materials (in an aqueous carrier) at a much higher concentration but in a much smaller volume than in the original contaminated water. This residue must be disposed of, however, which still may require transport to an acceptable facility. To be most effective, the regeneration system should strip the adsorbed material and convert it to a form suitable for disposal at the spill site.

In a prior Federally sponsored program (Juhola, A.J., "Laboratory Investigation of the Regeneration of Spent Activated Carbon", U.S. Public Health Service, Contract No. 14-12-469, Final Report MSAR 70-184), a laboratory-sized carbon regeneration unit was developed that used thermal treatment to strip the adsorbed material from the carbon. This unit was coupled to a gas-fired incinerator to provide high temperature thermal destruction of the stripped material. The laboratory unit served as a starting point for the design of a portable unit to provide onsite carbon regeneration and waste material incineration.

The goal of the current program was to design, construct, and test a full size mobile system for field regeneration of carbon. Several tasks were pursued toward this goal. These included:

(a) an in-depth assessment of existing carbon regeneration technology;
(b) selection of representative pollutants for which such a system might find use;
(c) preparation and evaluation of a preliminary design;
(d) laboratory-scale studies of the proposed design;
(e) modifications of the design into final specifications for fabrication;
(f) fabrication of a mobile system; and
(g) shakedown testing of the unit prior to transfer to the US EPA for further testing and field use.

The studies undertaken for each of these tasks are reported in the various sections of this report.

SECTION 2

RESULTS AND CONCLUSIONS

The program was successful in designing and fabricating a trailer-mounted carbon regeneration system based on design calculations and testing of a laboratory-scale system. The system consisted of a rotary kiln thermal regenerator, an incinerator or after burner to degrade the material stripped from the carbon, and a scrubbing tower to remove undesirable materials from the offgases.

The unit is self-contained for operation at a spill or hazardous waste site when deployed near a source of fresh water and provided with fuel. As constructed, the system is capable of regenerating 45.4 kg/hr of >40-mesh size granular carbon with <20% carbon loss and restoration of >75% of the adsorptive capacity.

Although indirect heating of such a kiln offers many advantages, weight and size restrictions forced the selection of direct firing for the kiln. Even then, an initial goal of a mobile system capable of regenerating 91 kg/hr of carbon could not be achieved and the size was reduced by 50%.

System design parameters were imposed by trailer limitations of size and weight and expected over-the-road stesses. Lightweight and resilient fabrication was emphasized throughout, including light structural containment, ceramic fiber thermal insulation, and flexible piping.

The system is constructed in a specially modified semi-trailer van that is 2.44-m-wide, 13.7-m-long, and 4.1-m-high when closed for storage or transit. When fitted for transport by tractor-truck or rail piggy-back, the van meets all applicable requirements, regulations, and conventional load limits.

Using the system and a carbon contaminated with Toxaphene and minor amounts of other chlorinated hydrocarbons, regeneration gave an 88% volume yield of a completely reactivated carbon based on iodine numbers.

A pilot run was made with an activated carbon material contaminated with toxaphene ($C_{10}H_{10}Cl_6$) and minor quantities of other chlorinated hydrocarbons Carbon loadings were 13% contaminant and 52% water. Regeneration gave an 83% volume yield of a completely reactivated material based upon iodine numbers. Vent gas analysis showed CO and hydrocarbons to be below detectable limits.

SECTION 3

PHASE I - PRELIMINARY SYSTEM DESIGN

The first phase of the program was directed to the development of a data base which would permit the design of a field scale portable carbon regneration system. It was divided into three tasks : a technology assessment of carbon regeneration, a review of adsorbate/carbon characteristics, and a preliminary system design.

TASK I - TECHNOLOGY ASSESSMENT

Work in this task was directed to the identification of those techniques which could be considered for carbon regeneration and management of the stripped waste and assessment of their suitability to a mobile system.

Based on the desired characteristics of a mobile carbon regeneration system and the expected behavior of the major categories of spill-chemicals during thermal regeneration, the major components of a basic system are given in the schematic flow diagram of Figure 1. This system is based upon the laboratory unit previously built by MSA and includes the following representative operations,

1. Carbon feed
2. Regeneration/activation
3. Carbon recovery and sizing
4. Condensation
5. Decantation/distillation or other method of concentration
6. Thermal incineration
7. Scrubbing and scrubber liquid treatment.

Of these major system operations, the regenerator/activator, is most critical. Other component requirements are dependent on the design and characteristics of the regeneration/activation operation. Illustrative calculations were made to define this dependency and to allow preliminary assessment of candidate systems.

Classification of Spill Chemical

Surveys conducted on spillage of polluting materials show a great variety of chemical substances that may become objects of water cleanup operations. The objective of this part of the study was to evaluate this great variety of chemicals relative to their ease or difficulty of

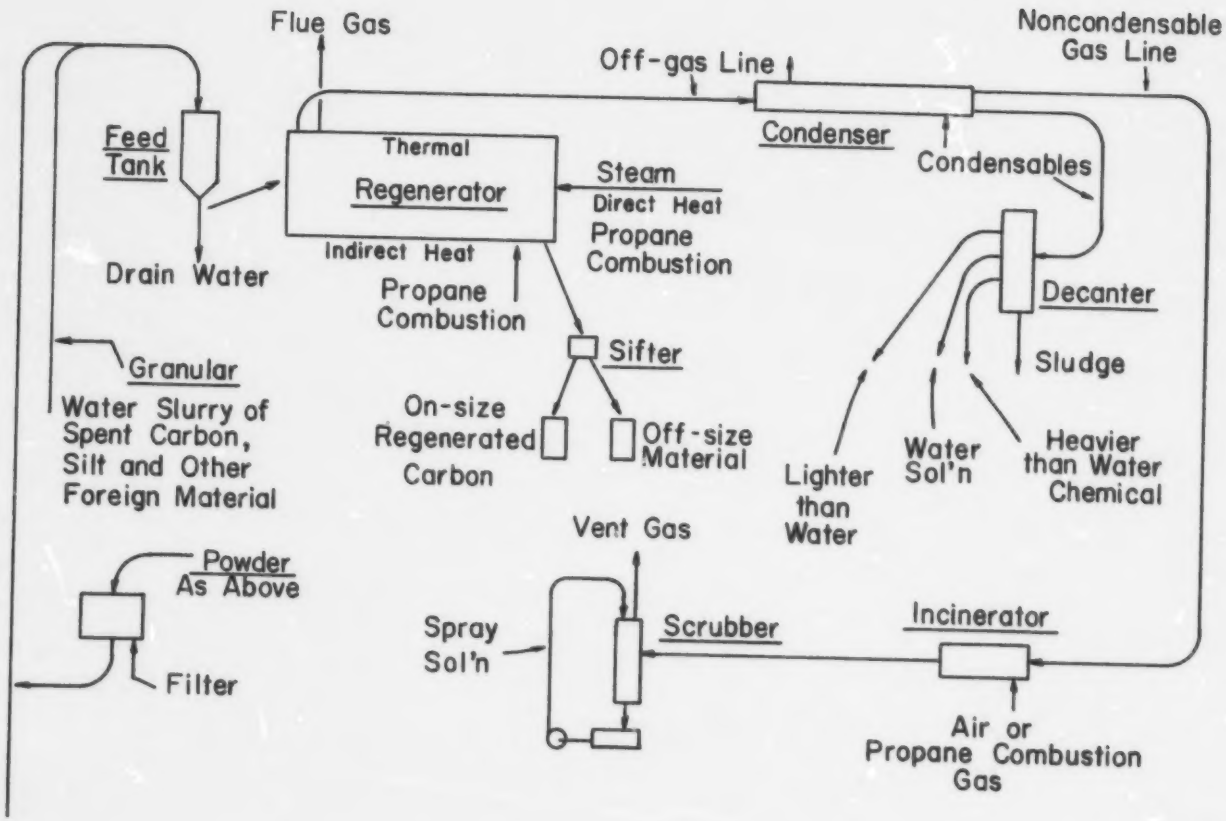


Figure 1. Flow diagram of proposed carbon regeneration system.

desorption from the carbon and to determine what steps must be taken to collect the desorbed chemicals and avoid further air or water pollution.

Spill chemicals were classified into five groups according to their distinctive regenerative patterns. Table 1 presents a partial listing of each group showing typical chemicals and range of properties.

Groups 1 and 2 are relatively volatile and have small molar volumes. Both properties correlate with relatively low to moderate adsorptive affinity, hence these two groups will be desorbed quite readily. At a 350°C final regeneration temperature, recovery of over 80% of the adsorptive capacity can be expected. Some decomposition of the heavier members of the groups can be expected to occur.

Group 2 differs from group 1 in that each chemical contains chlorine, sulfur or nitrogen. For group 1, an incineration step to convert the desorbed chemical to CO_2 and H_2O is sufficient to avoid air pollution. For group 2 a water or caustic scrubber downstream of the incinerator is required to remove the HCl, SO_2 or NO_2 from the exhaust gas stream.

Groups 3 and 4 are considerably less volatile than groups 1 and 2. Since they do not desorb readily, higher regeneration temperatures are required and considerable decomposition of the adsorbed chemical can be expected. One of the decomposition products will be free carbon. Its accumulation over a few adsorption-regeneration cycles can drastically decrease the carbon adsorptive capacity. To remove the free carbon, by steam or CO_2 oxidation, final regeneration temperatures near 930°C are required. Thus, for groups 3 and 4, a regenerator is required that can withstand a considerably higher temperature than that necessary for groups 1 and 2. Similar to groups 1 and 2, group 4 differs from 3 in that the chemicals contain chlorine, sulfur and nitrogen, requiring water or caustic scrubbing after incineration to avoid air pollution.

Group 5 resembles groups 3 and 4 in its regenerative behavior except that the group 5 chemicals contain nonvolatile metals and leave a metallic oxide or hydroxide residues. Alkaline metals form hydroxides and phosphorus forms an acid. These leach out to a large extent during the adsorption phase. Calcium, mercury, lead, copper, nickel and chromium form oxides or hydroxides which do not leach out. Mercuric chloride and lead acetate are strongly adsorbed, but are difficult to remove. Mercuric chloride sublimes at 270°C in the free state but may not when adsorbed. In this case, regeneration may not be beneficial. Lead acetate can be expected to decompose producing a lower lead oxide with a molar volume change from 100 to 28 cc/vol. After a second adsorption-regeneration cycle, further regeneration cycles may be of no benefit. Regeneration attempts on carbons containing copper sulfate, nickel chloride, chromic chloride and similar inorganic salts can be expected to be ineffective.

TABLE 1. CLASSIFICATION OF POTENTIAL SPILL CHEMICALS RELATIVE TO THEIR EASE OF DESORPTION FROM ACTIVATED CARBONS DURING REGENERATION

Group	Chemical	Density (g/cc)	Solubility in water (g/g at 20°C)	Liquid molar vol (cc/mole)	Vapor pressure (mm Hg at 25°C)
	Acetaldehyde	0.78		56	750
	Acetic acid	1.05		57	15
	Acrolein	0.86	0.4	65	270
	Allyl alcohol	0.85		68	24
	Propanone	0.79		73	170
1	Methyl acetate	0.97	0.33	76	160
	Propyl alcohol	0.78		77	19
	Benzene	0.88	0.0007	88	97
	2-Butanone	0.80	0.23	90	100
	Toluene	0.87	0.0005	106	28
	Pentane	0.63	0.0004	114	490
	Hexane	0.66	0.0001	130	150
	Acetonitrile	0.78		53	90
	Acrylonitrile	0.80	0.075	54	190
	Dichloromethane	1.33	0.020	64	400
	Carbon disulfide	1.26	0.0012	66	350
	Propanenitrile	0.77	0.10	71	45
	Dimethyl sulfide	0.85	insol.	73	480
	1,2,Dichloroethane	1.26	0.0087	78	220
2	Chloroform	1.49	0.008	80	130
	Allyl chloride	0.94	insol.	81	350
	Pyridine	0.98		81	18
	Propylamine	0.72	very sol.	82	240
	Propanethiol	0.84	slight	90	170
	Carbon tetrachloride	1.59	0.0008	97	105
	Diethylamine	0.71	very	103	230
	Ethylene glycol	1.18		62	198 BP
	Benzyl alcohol	1.04	0.038	108	205 BP
	Hydroquinone	1.32	0.012	110	258 BP
	Maleic acid	1.59	0.788	116	138 MP
	Benzoic acid	1.32	0.0029	122	249 BP
	Phenyl acetate	1.09	slight	136	266 BP
	Methylpentylacetate	0.87	slight	144	148 BP
3	Octanediol	---	slight	146	172 BP
	Carbitol	1.12	soluble	106	245 BP
	Butyl carbitol	0.96		162	231 BP
	Phthalic acid	1.59	0.0070	166	206 MP
	Ethylhesylacetate	0.87	insol.	172	199 BP
	Glucose	1.56	0.49	180	146 MP
	Sucrose	1.59	2.04	342	206 MP

(continued)

TABLE 1. (CONTINUTED) CLASSIFICATION OF POTENTIAL SPILL CHEMICALS RELATIVE TO THEIR EASE OF DESORPTION FROM ACTIVATED CARBONS DURING REGENERATION

Group	Chemical	Density (g/cc)	Solubility in water (g/g at 20°C)	Molecular weight (g/mole)	Boiling or melting point (°C)
	Ammonium acetate	1.17	1.48	77	114 MP
	Benzonitrile	1.01	slight	103	191 BP
	Benzamidine	---	soluble	120	80 MP
	Benzyl Chloride	1.10	insol.	126	179 BP
	Dichlorobenzene	1.30	<0.0005	147	179 BP
	Benzenesulfonic acid	---	soluble	158	525 MP
	Dicambia	---	slight	220	
4	2,4, D acid	---	slight	221	138 MP
	Ammonium lauryl sulfate	---	---	267	---
	Chlordane	1.57	insol.	248	322 BP
	Ethylenediamine-tetraacetic acid	---	slight	292	---
	Diazinon	---	---	304	---
	DDT	---	insol.	334	108 MP
	Aldrin	---	insol.	364	104 MP
	Trimethyl phospate	1.25	very sol.	140	197 BP
	Calcium acetate	---	0.37	158	---
	Mercuric chloride	5.44	0.069	271	302 BP
	Solium stearate	---	sol	306	---
	Mercuric thiocyanate	7.56	0.0007	317	---
	Lead thiosulfate	5.18	0.0003	319	---
	Lead thiocyanate	3.82	0.00005	323	---
5	Lead acetate	3.25	0.44	325	280 MP
	Hexadecyl sulfate, sodium salt	---	soluble	344	---
	Mercuric sulfate	7.56	0.0006	497	---
	Zinc phenyl sulfate	---	0.655	556	---
	Potassium oleate	---	soluble	603	95 MP

Review of Potential Regeneration Systems

Regeneration of activated carbon requires the removal of the sorbed material. Three methods are available; thermal, solvent extraction and vacuum. The thermal method is the most widely used.

Solvent extraction uses leaching by acids, bases or solvent. It requires either chemical reaction and product solubility in the aqueous solution or high solubility in the solvent. Of the great variety of potential spill-chemicals, only a small portion can be effectively desorbed by this method. In addition, the leaching agent leaves the carbon in a spent condition and hence must in turn be removed with water (for acids or bases) or with heat or vacuum (for solvents).

Vacuum desorption is effective for some hard-to-desorb chemicals if the carbon is relatively dry and some heat can be applied. It is assumed in vacuum desorption that the deposited species can be removed by lowering the ambient pressure with no decomposition in the desorption process. The high water content in the carbon imposes a heavy load on the vacuum system. The process is usually restricted to regeneration of carbons used to purify gases.

Thermal regeneration offers the greatest versatility. It is able to cope with a wide range of materials from those easy to desorb to those most difficult to desorb, including those that may decompose in place. The former can often be vaporized along with the water. The other materials involve pyrolysis of the sorbed material, converting them to a volatile portion, and a carbon residue. To control the carbon, pyrolytic regeneration is performed at temperatures as high as 871°C. The carbon residue is oxidized to CO and hydrogen by means of steam or carbon dioxide. A diluent gas is often necessary to prevent excessive loss of the base carbon material when exposed to these reactivation conditions.

Thermal regenerators can be designed to cover a range of desorption conditions with simultaneous activation. Equipment varies primarily in the way in which heat is applied (direct or indirect firing) and the way in which the carbon is physically moved through the heated zone (rotary tube, multiple hearths or fluidized bed). There are specially designed reactors that use microwave heating; or wet air oxidation (the Zimpro process). The latter two are developmental and not commercially proven.

The rotary tube furnace or kiln utilizes internal vanes that lift the carbon part way up the wall, where it then falls back to the bottom. This effects a stirring condition while the carbon moves through the tube. Indirect firing involves heating on the outside. It has relatively poor heat transfer and a more bulky construction than a direct-fired unit where hot gases are passed directly through the tube and directly contact the carbon. As the carbon passes through the tube, it is dried in the first portion, regeneration is completed in the central portion and reactivation (if necessary) occurs in the latter protion. Temperature regimes are varied to meet the requirements for removal of specific sorbed materials.

A multiple hearth furnace has a vertical stack of platforms with wiping arms. The carbon on each platform is pushed toward an opening where it drops to the next lower level. The platforms are alternately arranged so that carbon moves from the center to the edge of one platform and from the edge to the center of the next lower platform. Gases pass upward through the furnace. Thermal distortion of the multiple hearths is a known problem. For that reason, this type of construction would not appear to be attractive for a mobile unit.

In a fluidized bed, streams of gas and carbon flow countercurrently. The carbon tends to float as a layer in the gas. Fluidized bed processes provide excellent heat transfer and a minimum of seals and moving parts compared to the rotary tube and multiple hearth units. A fluidized bed is a high energy consumer because of the increased pressure drop involved in maintaining a fluid bed condition. There is also the possibilty of increased attrition of the granular material, necessitating particulate removal equipment downstream. Multiple beds may also be required to provide the appropriate zones and conditions for drying, regeneration and reactivation. Although some development work has been done, there has been no commercial application of fluidized bed techniques to carbon regeneration/reactivation.

Treatment of the offgases will probable be required regardless of the type of regenerator used. There are differences to be recognized between the direct and indirect firing units with respect to such treatment. An indirect fired unit adds only heat to the interior of the system, and offgases are evaporated moisture and desorbed pollutants only. To help control the process, a sweep gas would most probably be required. The sweep gas could be steam, giving a condensable material that could be removed prior to treament of desorbed pollutants or their decomposition products. In direct firing, the flue gas will contain a significant amount of non-condensables (CO, CO_2, N_2, some O_2) that will add to the total gas that must pass through the treatment system after condensation of steam.

On the basis of the preliminary review, thermal activation clearly appeared to be the only logical choice, with the further conclusion that only rotary type units could be built on a scale small enough to fit the size and weight limitations of an over-the-road trailer.

Two basic designs were considered -- a rotary kiln and a rotary hearth furnace. Initially there was interest from manufacturers of rotary hearth furnaces to investigate design modifications which would allow trailer mounting. With time, however, it became clear that if modifications were possible at all, they would take considerable time and effort. Rotary kilns appeared better able to meet near term requirements and the program was directed to that option.

Direct and Indirect Heating

To evaluate the potential of direct and indirect firing modes on system design, preliminary calculations were made for four modes of operation. For

these calculations, a process throughput of 91 kg/hr (dry carbon basis) was assumed. These modes of operation are defined as follows:

1. Combination of direct-fired regenerator and carbon loaded (saturated) with groups 4 and 5 chemicals.

2. Combination of indirect-fired regenerator and carbon loaded with groups 4 and 5 chemicals.

3. Combination of direct-fired regenerator and carbon loaded with groups 1, 2 and 3 chemicals.

4. Combination of indirect-fired regenerator and carbon loaded with groups 1, 2 and 3 chemicals.

In mode 1 high temperatures up to 927°C are required to give an active carbon product. Three reactions occur: (1) vaporization of the water; (2) pyrolysis of the chemical, releasing a volatile product and depositing free carbon and (3) steam oxidation of the free carbon (steam activation).

In mode 2, temperatures and reactions are the same as in mode 1 except that indirect firing considerably reduces the volume of gases that are emitted from the regenerator. As a result, the downstream components of the system are smaller with more efficient condensation recovery.

In mode 3, lower temperatures are used, the maximum being about 316°C. In this case only vaporization of the water and desorption of the chemical are involved. The large volume of flue gas from direct firing limits the efficiency of condensation recovery.

In mode 4, the temperature and reactions are the same as mode 3 except that with indirect firing, the volume of noncondensable gases in the regenerator effluent is very small. When the incoming carbon slurry is clean (i.e. it contains only the carbon, clean water and the adsorbed chemical), the operating conditions can be very close to those used in solvent recovery. A virtually complete recovery of the chemical is possible and the incinerator and scrubber may not be necessary to the process.

System Functions

The various functions the system is required to perform are addressed in the following paragraphs in the sequence in which they are performed.

Carbon Feed--

The spent carbon is delivered to the system as a 5% to 30% carbon-water slurry. To reduce the water load on the regenerator, excess water is first removed by mechanical procedures.

Granular carbon slurry can be extracted by transferring the slurry into the feed tank which has provisions for drainage. Drainage reduces water content to about 40%, or 60% by weight dry carbon. The drained carbon is then

fed into the regenerator by an upward inclined screw feeder that allows further drainage during feeding.

For design purposes, the feed rate to the regenerator is:

Carbon (granular)	- 91 kg/hr
Adsorbed chemical	- 9.1 kg/hr
Water	- 65.8 kg/hr
Other adsorbates	- variable but small
Silt	- variable, unknown

Powdered carbon as a feed would consist of 10% or less of carbon as an aqueous slurry. In order to make carbon manageable in a screw feeder, the water content of the mixture would have to be reduced from 90% to 60% or lower. This can be done by centrifuging or filtering and drawing air through the filter cake. The dried centrifuge or filter cake can then be broken up and fed as lumps into the regenerator.

For design purposes, the feed rate would be:

Carbon (powdered)	- 91 kg/hr
Adsorbed chemical	- 9.1 kg/hr
Water	- 150 kg/hr
Other adsorbates	- variable but small
Silt	- variable, unknown

Details on powdered carbon feed are given here because evidence from MSA work on carbon spent in tertiary sewage treatment (Juhola, 1970) showed that powdered carbons could be regenerated in a modified indirect-fired rotary tube.

Regeneration--

The regenerator should perform the following functions and must perform the first three:

a. Vaporize water from carbon;

b. Desorb the adsorbed chemical if group 1, 2, and/or 3;

c. Pyrolyze the adsorbed chemicals if group 4 and/or 5;

d. Desorb group 1, 2, or 3 with minimum loss in recovery and pyrolyze group 4 or 5 in a mixture of group 1, 2, or 3 with group 4 or 5 present; and

e. Reactivate carbon with loss of carbon not over 10% or activity not over 30%.

Material and heat balance calculations were made for two situations: (1) only group 1, 2 or 3 chemicals are present and completely desorbed (Table 2), and (2) only group 4 or 5 chemicals are present with carbon reactivated to 100% original activity at 5% carbon weight loss (Table 3).

TABLE 2. MATERIAL AND HEAT BALANCE FOR GRANULAR CARBON REGENERATION AND ADSORBATE RECOVERY

Material	kg/hr	°C	Kcal/hr	Heat %
INPUT				
Act. carbon	91.0	16	---	
Adsorbate	9.1	16	---	
Water	65.8	16	---	
Steam sweep	22.8	104	832	13·3
ΔH= -1060 cal/mole	(22.8)	--	13,457	
Propane	7.8	16	---	
ΔH= - 21,700 cal/mole	(7.8)	--	93,492	86.7
Air	126.6	16	---	
	323.1		107,781	100.0
OUTPUT				
Act. carbon	91.0	316	9,551	8.9
Steam from carbon	65.8	149	3,956	40.0
ΔH= +1060 cal/mole	(65.8)	--	39,060	
Desorbed vapor	9.1	149	302	
ΔH= +2760 cal/mole	(9.1)	--	13,910	27.0
Steam sweep	22.8	149	1,336	
ΔH= +1060 cal/mole	(22.8)	--	13,457	
Heat loss, 10%			8,064	
Propane flue gas				
N_2	93.4	316	7,409	24.0
CO_2	23.0	316	1,109	
H_2O	12.7	316	1,688	
ΔH= +1060 cal/mole	(12.7)	--	7,510	
	322.8		107,352	99.9

For the adsorbate recovery operation (Table 2), the following operating temperatures were assumed to be adequate to accomplish the desorption of the chemicals:

Heat Source	Temp. (°C)
Base (ambient)	16
Steam input	105
Act. carbon output	316
Effluent steam and chemical	149

Based on the data shown in Table 2 and the assumed feed composition, about 40% of the heat requirement is for water desorption and 27% for chemical desorption.

The heating requirements for regeneration by desorption only can be met by direct heating or indirect heating. The means of heating affects the offgas flow, as shown in Table 3:

TABLE 3. EFFECT OF DIRECT AND INDIRECT HEATING ON GAS FLOWS FOR THERMAL DESORPTION

Gas	Flow (kg/hr) Direct	Indirect
Steam from carbon	65.7	65.7
Chemical desorbed	9.1	9.1
Steam sweep	--	22.7
Combustion steam	12.7	--
Combustion CO_2	23.1	--
Combustion N_2	98.4	--
Totals	209.0	97.5

When the heating is direct, the offgas flow to the condenser is about 209 kg/hr. Of this, 121.5 kg/hr are noncondensable CO_2 and N_2. These gases will continue downstream and carry with them considerable amounts of group 1 and 2 chemicals. When group 2 chemicals are present, incineration becomes a necessary next step to convert the chlorides along with nitrogen and sulfur compounds to a form that can be scrubbed out ot the gas stream by water or caustic solution.

When heating is indirect, the offgas flow to the condenser is 97.5 kg/hr. If the gas system is devoid of foreign noncondensable gases, virtually all of the chemical and water would be condensed with very little or no gas going to the incinerator.

When group 4 or 5 chemicals are present, pyrolysis and reactivation are part of the regeneration process. Materials and heat balance for this situation are shown is Table 4. In this case, because of higher temperatures, a 45% carbon loss is assumed. The operating, temperatures noted below were assumed to be necessary to accomplish pyrolysis and carbon regeneration.

TABLE 4. MATERIAL AND HEAT BALANCE FOR GRANULAR CARBON REACTIVATION

INPUT

Material	kg/hr	°C	Kcal/hr	Heat %
Act. carbon	91.0	16	---	
Adsorbate ($-CH_2-$)	9.1	16	---	
Water	65.8	16	---	
Steam for acct.	22.8	104	832	8.9
ΔH= -1060 cal/mole	(22.8)	--	13,457	
Propane	12.1	16	---	
ΔH= - 21,700 cal/mole	(12.1)	--	145,656	91.0
Air	199.0	16	---	
	399.8		159,945	99.9

OUTPUT

Act. carbon	86	899	34,272	21.5
Steam from carbon	65.8	316	8,820	30.0
ΔH= +1060 cal/mole	(65.8)	--	39,060	
Desorbed gas ($-CH_2-$)				
ΔH= +2760 cal/mole	(5.8)	---	8,820	
CO	11.4	316	857	
H_2	1.6	316	1,462	
Free carbon				
ΔH= +4800 cal/mole	(3.3)	--	8,870	
CO	7.8	316	580	23.3
H_2	0.5	316	479	
Act carbon loss				
ΔH= +4800 cal/mole	4.5	--	12,096	
CO	10.6	316	781	
H_2	0.8	316	680	
Excess Act steam	3.8	316	504	
ΔH= +1060 cal/mole	3.8		2,092	
Heat loss, 7.5%			11,844	
Propane flue gas				
N_2	154.2	316	11,592	
CO_2	35.8	316	2,520	25.1
H_2O	19.5	316	2,621	
ΔH= +1060 cal/mole	(19.5)	--	11,592	
	402.3		159,542	99.9

Heat Source	Temperature (°C)
Base (Ambient)	16
Steam input	105
Act. carbon output	899
Effluent gases	316

In this case per Table 4, 30% of the heat requirement is for water desorption and 23% for chemical pyrolysis and carbon reactivation. It also assumes a 5% carbon loss.

For pyrolysis and activation, as was the case when desorption was the only concern, the heating requirements can be met by direct or indirect methods. The affect on the amount of offgas flows is shown in Table 5:

TABLE 5. EFFECT OF DIRECT AND INDIRECT HEATING ON GAS FLOWS DURING PYROLYTIC REGENERATION

	Flow (kg/hr)	
Gas	Direct	Indirect
Steam from carbon	65.7	65.7
Chem. decomp. products		
CO	19.1	19.1
H_2	2.3	2.3
Act. carbon loss (5%)		
CO	10.4	10.4
H_2	0.9	0.9
Excess steam	--	3.6
Combustion steam	19.5	--
Combustion CO_2	35.8	--
Combustion N_2	154.2	--
Totals	307.9	102.0

When the heating is direct, the offgas flow to the condenser is about 308 kg/hr. Of this, 218 kg/hr are noncondensable gases (CO_2, CO, H_2 and N_2) and will pass through the condenser. For ease of calculation, it was assumed that the gaseous pyrolysis products reacted with the steam forming CO and H_2. In actual practice, this happens only partially. If NO_x, SO_x or chlorides are present in the adsorbed chemical, these groups would be partially emitted as inorganic compounds. An incinerator and scrubber are needed downstream to strip them from the vent gas.

When the heating is indirect, the offgas flow to the condenser is about 102 kg/hr, of which 32.7 kg/hr is noncondensable and combustible CO and H_2. Any NO_x, SO_x or chlorides would be partially or completely decomposed to release either the inorganic acid gas or organic decomposition product which must be further decomposed in the incinerator.

Powdered carbon will have a water content over twice as large as that for the granular carbons. The heat requirement will be correspondingly higher, but both desorption and reactivation will require much milder conditions. It can be expected that the regeneration condition would not be much greater than those for the granular carbons. Powdered carbon is not often used in wastewater treatment because of high physical losses in air and water streams.

The four ways of carrying out the regeneration process will have a bearing on the size of the condenser and other components downstream of the condenser as shown in Table 6.

TABLE 6. EFFLUENT GASES FROM REGENERATORS OPERATED UNDER DIFFERENT CONDITIONS

Mode	Process	Condensable (kg/hr)	Noncondensable (kg/hr)
1	Direct heat, adsorbate pyrolysis and carbon reactivation	86.2	222.3
2	Indirect heat, adsorbate pyrolysis, and carbon reactivation	68.0	31.8
3	Direct heat and adsorbate desorption only	86.2	121.7
4	Indirect heat and adsorbate desorption only	95.3	nil

Gases from modes 1 and 2, pyrolysis and reactivation, leave the regenerator at 315°C and those from modes 3 and 4 at about 149°C. For powdered carbon, the condensable input rate to the condenser would be over 150 kg/hr, primarily water.

Indirect Fired Rotary Kiln--

The most favorable conditions for spilled chemical recovery/detoxification and control of air and water pollution appeared to lie with an indirect fired regenerator. With indirect firing, the combustion gases are not contaminated by the adsorbate or its decomposition products and thus can be emitted directly to the atmosphere (assuming propane or other clean burning fuel) without further treatment. The contaminated gas stream consists only of desorbed materials, steam and a small volume of sweep gas (which may be steam or flue gas). This allows recovery by condensation and incineration/scrubbing of a small volume of non-condensibles or, alternatively, incineration/scrubbing of the total stream with minimally sized equipment.

Indirect fired rotary kilns are employed in a variety of industrial operations and design data are readily available. The major design criteria are the kiln areas and volume required for efficient heat transfer and the solids loading, as percent of total volume. In industrial practice, volume loading ranges from about 2 or 3% to perhaps as high as 15%, depending on the moisture content and flow properties of the solid being handled. Based on experience with rotary tube regeneration, a loading of 10% appears feasible for granular carbon having a drained moisture content of about 40%.

At 10% loading, the minimum size of rotary kiln that could handle both regeneration and activation of 91 kg/hr carbon (dry basis) would be a tube of 0.6 m inside diameter with a 4.6 m heated length. From discussions with manufacturers of this type of equipment, it was estimated that a complete kiln of this throughput would have overall dimensions of 2.1 m x 2.1 m x 10.7 m and would weigh approximately 22.7 kkg (25 tons). Estimated minimum delivery time was about one year.

This is both too heavy and too large to fit on the semitrailer and still leave work space and room for the other components of the system. It was apparent that either the regeneration rate would have to be drastically reduced from the 91 kg/hr rate or chemical recovery would have to be sacrificed. Since the primary objective is to reduce environmental damage utilizing carbon adsorption the correct decision appears to be to maintain the regeneration rate level as high as possible. (Chemical recovery is of secondary importance).

Direct Fired Rotary Kiln--

Direct fired kilns can be operated at higher throughputs than can be attained with indirect fired units of similar dimensions because of more efficient heat transfer. For this reason, direct fired kilns have more sizes and designs commercially available.

Two quotations were obtained from a well known manufacturer; one for a 91 kg/hr (dry) carbon and one for a 45.4 kg/hr carbon system. Both are packaged systems consisting of the rotary kiln proper, dewatering feed screw, afterburner, offgas scrubber, and quench tank. The smaller kiln with a 380 mm inside diameter refractory-lined tube 3.80 m long, appeared capable of being fitted onto a semitrailer with all the other required equipment.

In view of the space limitations, the smaller (45.4 kg/hr) direct fired rotary kiln was selected as most appropriate for further analysis.

Incinerator--

Noncondensable gases entering the incinerator or afterburner are heated to 760° C for 0.5 sec in an oxidizing atmosphere. The size of the incinerator can vary considerably depending on the regeneration processes used upstream. Of the 222 kg/hr of noncondensable gases produced in mode 1, about 15% will be combustible gases, consisting of spill chemical, CO and H_2. Of the 31.8 kg/hr, mode 2, most is combustible with a high percentage of CO and H_2. Of the 121.7 kg/hr, mode 3, only about 3% will be combustible gas. In each case,

air with or without supplementary heating is required to raise the temperature to 760°C to oxidize the gases.

Table 7 gives estimates of total final gas volume in the incinerator at 760°C STP and anticipated interior volume when residence time is 0.5 sec.

TABLE 7. INCINERATOR SIZE ESTIMATES FOR 0.5 SEC RESIDENCE TIME AT 760°C

Process No.	Gas Volume m^3/min at STP	Gas Volume m^3/min at 760°C	Incinerator Volume m^3
1	13.3	50.4	0.4
2	5.0	19.0	0.2
3	2.5	10.7	0.1

In calculating the gas volumes for modes 2 and 3, it was assumed that about one half of the desorbed chemical passes through the condenser because of the high volume of noncondensable gases. For heating calculations, the desorbed chemical was assumed to be composed of $-CH_2-$ groups. In each process, it is also assumed that enough combustibles are present to make the incinerator self supporting, i.e., no supplementary heat is required, even if the incinerator has to operate at temperatures above 760°C. At the postulated temperatures and 0.5 sec residence time, it is expected that well over 90% of the gases will be decomposed to CO_2 and H_2O, and HCl, NO_2, and SO_2 if chlorine, nitrogen, and sulfur are present.

The probable amount of CO_2 emitted from each mode, assuming 50% of the organics in modes 1 and 3 are condensed out, will be as follows:

Mode 1 - 82.1 kg/hr
Mode 2 - 46.3 kg/hr
Mode 3 - 37.2 kg/hr

Scrubber--

The combustion products from the incinerator are passed through a scrubber containing water or a 20% caustic solution to remove any HCl, NO, SO_2 or other acid gas present. The gas volumes entering the incinerator will be as given in column 2 or 3 in Table 7.

TASK II - PRELIMINARY SYSTEM DESIGN

Design studies were concentrated on a direct fired rotary kiln system. The preliminary design calculations for carbon regeneration/activation were based on a system throughput of 45.4 kg/hr (dry regenerated carbon), assuming 5% carbon loss in processing. The carbon feed was assumed to have an average moisture content of 40% and an adsorbate loading of 0.1 kg per kg of carbon. A commercial kiln designed to handle 45.4 kg/hr of carbon for sugar purification was used as the basis for design. By modifying the equipment arrangement, a suitable spatial arrangement was devised to allow trailer

mounting. A schematic of this arrangement is shown in Figure 2. The incinerator is mounted directly above the kiln tube for space saving.

Calculations based on the capacity of the kiln tube size (380 mm inside diameter (ID) x 3.80 m length) would indicate the feasibility of handling more than 45.4 kg/hr of granular carbon. Heat transfer considerations may, however, limit operation to near design capacity. Availability data on direct fired heat transfer are not sufficient to allow accurate prediction. Major factors such as initial moisture content and ease of regeneration (final temperature requirements) will significantly affect throughput.

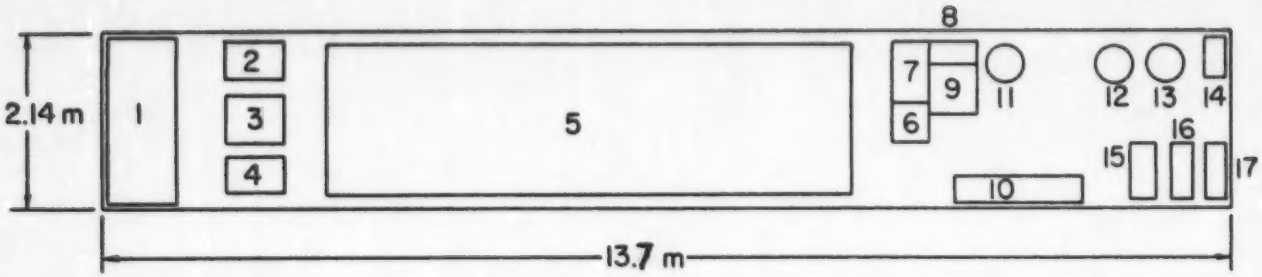
A rotary tube of 380 mm by 3.80 m long will provide a holdup in the range of 10 to 15% of tube volume, and retention times in the range from 0.5 to 0.75 hr. Previous data on granular carbon regeneration/activation indicated these retention times are sufficient for complete reactivation. Retention times and holdup can be varied however by adjustment of tube slope and rotation rates.

After the kiln size and system layout were defined, regeneration/activation process design calculations were completed for several different cases of operation of a direct fired rotary kiln. The calculation summary for two of these cases is given in Table 8. The major factor in process requirements and sizing of downstream components (incinerator, etc) is the physical form of the water required to cool the hot furnace gases and provide steam for activation. Lower fuel requirements, gas velocities and gas flow rates are achieved by injecting liquid water rather than steam. Both heat of vaporization and the sensible heat of the steam can be utilized.

Case I conditions, liquid water injection, were selected for continued design development. The incinerator required for treatment of the kiln off-gas at 982°C and 0.5 sec residence time was calculated to be 0.23 m^3 in volume, 305 mm inside diameter by about 3.4 m long.

Manufacturer's data on lightweight, high temperature insulation were reviewed and kiln tube weight tradeoff calculations were made. Alumina-silica fiber insulation with desirable heat transfer and mechanical properties is available in both standard and custom fabricated shapes. At a bulk density of 0.45 g/cm^3, this insulation provides considerable weight savings over clay-based refactories with densities of the order of 2 g/cm^3. A 76 mm layer of this type of insulation enclosed within a 457 mm diameter steel pipe was estimated as being adequate to insulate and allow mounting the incinerator above the kiln.

Using these design calculations along with weights of the supporting equipment, a weight analysis was made. The values employed are given in Table 9. The total weight of 42,185 kg is clearly in excess of the 33,113 kg trailer limit for over the road operation without special permits. Consequently, the design was reviewed to find areas where weight could be reduced and/or eliminated. These areas were identified as the kiln feed, the scrubber and kiln barrel accessories.



Equipment

1. Generator (electric)
2. Slurry pump
3. Dewatering screw
4. Cond. water pump
5. Kiln
6. Quench tank
7. Elevator
8. Dryer
9. Screener
10. Panel board
11. Steam generator
12. Still
13. Scrubber
14. Scrubber pump
15. Still feed pump
16. Transfer pump
17. Transfer pump

Figure 2. Mobile Regeneration Unit (45.4 kg/hr Carbon).

TABLE 8. PROCESS CALCULATION SUMMARY*

Item	Case I offgas at 315°C water added	Case II offgas at 315°C steam added
Propane Required, kg/hr	10.8	40.2
Inlet Gas Velocity, m/sec	2.7	13.7
Heat Load, kcal/hr	129,600	485,000
Offgas Flow Rate, m^3/hr	606	2,566
Offgas Velocity, m/sec	1.5	6.2
Steam or Water Added, kg/hr	49.0	420

*Basis: 45.4 kg/hr carbon output; 2.3 kg/hr carbon loss; 4.5 kg/hr adsorbate; 40% water content in feed; activation temperature 899°C; kiln furnace temperature 1093°C

TABLE 9. ESTIMATED WEIGHT FOR MOBILE CARBON REGENERATION UNIT WITH DIRECT FIRED ROTARY KILN

Item	Estimated Weight (kg)
Diesel Generator, 50 Kw	1,814.4
Rotary Kiln, 380 mm (I.D.) x 457 mm (O.D.) x 3.80 m (Long) Dewatering feed screw Afterburner/Incinerator Offgas scrubber Carbon quench tank	11,340
Process Equipment Elevator dryer Screener Auxiliary scrubber Still Pump (5) Control panel	5,443.2
Misc. Equipment Steam generator Holding bags and hose Trolley hoists and winches Lighting and ventilation Piping (material) Fire and safety equipment Special framing, walkways, etc.	11,340
Tractor	7,257.6
Trailer, 13.7 m long	4,989.6
Total	42,184.8

The initial kiln feed system proposed for handling slurries or wet feeds employed a dewatering screw feeder. Consideration of potential field situations, volumetric feed rates, and granular carbon drainage rates, showed the possibility of completely eliminating the requirement of a dewatering screw feeder. The initial draining of the carbon feed could be achieved external to the trailer mounted system. The drained carbon could then be transferred to a feed system on the trailer. This would consist simply of a feed hopper and associated feed screw to transfer the drained carbon to the kiln. Model studies indicate this modified hopper and feed screw would extend only about 1 m beyond the feed housing of the kiln versus 3 m for the dewatering screw.

An investigation of the jack arrangement which provides adjustment of the kiln barrel slope was made, with the decision that it could be eliminated. The nominal slope had been set at 208 cm/m with adjustment possible both in slope and speed to vary conveying rate. The analysis showed that sufficient variation could be provided by the adjustment of rotational speed alone, with some adjustment possible by shimming the channel supports.

A design review was also made on the scrubber water system to investigate weight savings. Ion exchange treatment of total process makeup water was considered first but exchange resin requirements were deemed to be prohibitive in both cost and size. The design selected employs continuous caustic treatment and filtration of all makeup water, with scrubber recycle through an air-cooled heat exchanger to minimize makeup requirements. Process calculations considered both normal and anticipated maximum water requirements. Maximum makeup water required for the kiln and scrubber system is 8.3 l/min.

Caustic treatment of both makeup water and scrubber recycle and waste streams will require a maximum of 45 kg/day of NaOH or 30 l/day of 50% caustic. This assumes a maximum acid gas formation (as HCl) from adsorbate incineration. Normal operation may produce less acid and lower caustic treatment requirements. Additional water makeup is required for product carbon cooling. Wet processing of the product increases the total water makeup requirements to 19 l/min.

Changes in the three areas noted plus a variety of small modifications enabled a reduction in the estimated equipment weight to 18,144 kg. This allows for a standard trailer configuration without the need for special operating permits. The final design and spatial arrangement were as shown schematically in Figure 3.

TASK III - BENCH SCALE STUDIES

A number of the decisions to be made in the delineation of the system design depended on an understanding of operating requirements. To provide this particular data, a series of studies were conducted using the laboratory

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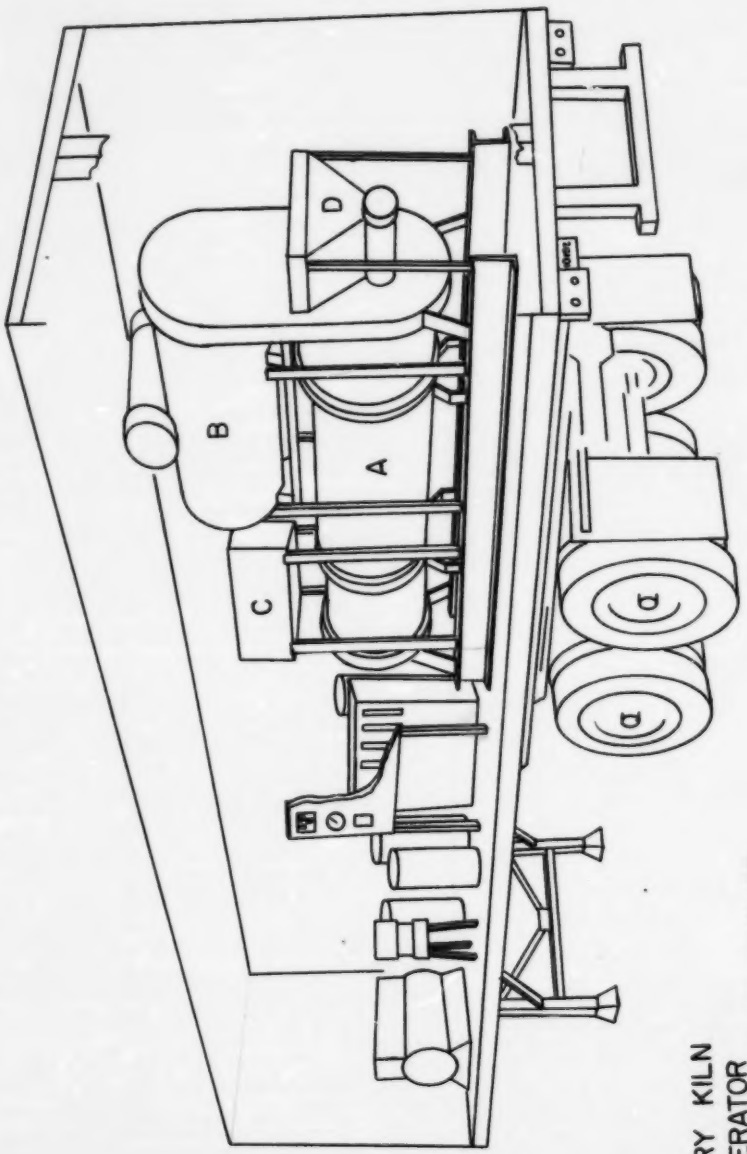

A. ROTARY KILN
B. INCINERATOR
C. SCRUBBER - LIQUID COOLER
D. CARBON FEEDER

Figure 3. Trailer design

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(PREVIOUSLY CIN. EPA FORM 2571)
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TYPING GUIDE SHEET

scale regeneration unit built during the earlier EPA program. These studies had two objectives: an evaluation of the regeneration/incineration design of the proposed mobile unit, and the testing of certain proposed materials of construction under simulated conditions.

Bench Scale System

The design and operational characteristics of the laboratory-sized regenerator are given in the final report, "Laboratory Investigation of the Regeneration of Spent Activated Carbon" (Juhola, 1970). The unit was built as an indirect fired system. The regenerator is an electrically heated rotary (stainless steel) tube 82.5 mm in diameter with a 1080 mm heated length. An auger type volumetric feeder is used to meter the carbon into the rotary tube. To approximate the direct firing characteristics of the proposed regenerator/incinerator, a countercurrent flow of gas was added to the laboratory unit to simulate the products of propane combustion.

The laboratory regenerator operates at 18% loading with a .590 kgm/hr (regenerated) carbon throughput. The exhaust gas cleanup train consists of a thermal incinerator, an air-cooled condenser, and a water spray scrubber. The thermal incinerator is designed to operate at 900°C, and gives a 0.5 sec residence time to the effluent gas from the regenerator. It is heated with natural gas at startup, the flow of which is reduced as the concentration of desorbed flammable products increases and comes to a steady-state flow rate. Air input is metered to maximize combustion efficiency.

The condenser is a finned tube and gives about 0.5 sec residence time to the gases from the incinerator. Some 25 wt % of the gas is water vapor and separates from the noncondensable gases, N_2 and CO_2, in the scrubber. The condenser reduces the heat load on the scrubber and minimizes the amount of scrubber liquid which needs to be recirculated. The cleanup train is arranged for operation with or without the scrubber or condenser in the train. In this way, data can be obtained on various alternate routes for exhaust gas cleanup.

The CO_2, N_2, air and part of the gas stream are metered in to simulate the products of combustion of propane in air. Extra steam is added by a steam generator to simulate moderation of the flue gas temperature. The water from the carbon and most of the chemicals are desorbed before the carbon reaches the hot zone of the rotary kiln. This mixture of input materials approximates the proportions that will be seen by the mobile unit.

Chemical Classification and Selection

Evaluation of the regeneration/incineration capabilities of the proposed mobile system required the selection of representative chemicals from each of the five categories previously identified. Each group has specific properties which effect its behavior in the thermal cycle.

For each of the five groups of spill chemicals, representative materials were chosen for study in the bench scale rotary kiln. The following are the selected test chemicals for each group.

Group	Representative Chemical	Mol. Wt. (g/Mole)
1	Methyl acetate	74
2	Dichloromethane	85
3	Diethyleneglycol diethyl ether	162
4	Benzenesulfonic acid	158
	Hexamethylene tetramine	140
5	Tris(2-chloroethyl)phosphate	277

Regeneration/Incineration Tests

Regeneration runs were made with each of the five materials. Three consecutive runs were made on each of the chemicals except for hexamethylene tetramine, using Pittsburgh CAL carbon. The hexamethylene tetramine runs were not completed because of corrosion problems in the incinerator.

In preparing the spent carbons for the tests, the carbon was treated with 10% by weight of chemical and 50% by weight of water. For water soluble materials the additions were made as aqueous solutions. For water insolubles, the chemical was added first and then the water. In each case, the mixture was allowed to stand overnight to allow equilibration of the chemical with the carbon.

For the dichloromethane runs, the feed rate was 1.26 kg/hr, giving a residence time of 45 minutes and a volumetric loading in the rotary tube of about 16%. When the necessary flows of N_2, CO_2, O_2 and steam to simulate direct firing were fed to the rotary kiln, the gas velocity was such that considerable quantities of carbon dust were carried downstream to the incinerator and scrubber. In the runs for the other chemicals, the carbon feed was decreased to 0.72 kg/hr, giving a volumetric loading of about 9% for a 45 minute residence time. This alleviated the problems generated by carbon dust carryover.

Satisfactory results were obtained in all test runs. The methyl acetate and dichloromethane were completely decomposed on passing through the incinerator. The incinerator was found to operate best between 816°C and 871°C with an air flow of 18 l/min. The HCl formed from the dichloromethane was removed from the gas stream along with the steam condensate by the air cooled condenser. The scrubber was not used in these tests. HCl concentrations in the condenser effluent gas stream ranged from 10 to 60 ppm.

Similar tests were carried out for the other chemicals. Typical concentrations of gases from the regenerator and scrubber for each of the other test chemicals are given in Table 10. Some fog appeared in the vent gas for the benzene sulfonic acid and tris(2-chloroethyl)phosphate runs.

TABLE 10. TYPICAL CONCENTRATION OF GASES EXITING FROM THE LABORATORY SCALE REGENERATOR AND SCRUBBER

Test Chemical	Exit Gas Concentration (ppm)	
	Regenerator	Scrubber
Benzenesulfonic acid	CO - >2000 HC*- >2000 SO_2 - >400 H_2CO - 30	CO - 50 HC - none H_2CO - none SO_2 - 30 H_2CO - 5
Diethyleneglycol diethyl ether	CO - 4000 CH_3COOH - >1000 H_2CO - 100	CO - 35 CH_3COOH - none H_2CO - none NO - 6 NO_2 - none
Tris(2-chloroethyl)phosphate	CO ->4000 HCl - 300 NO - none CH_3COOH - 27	CO - 45 HCl - 90 CH_3COOH - 24

*HC = Hydrocarbon

Apparently the SO_2 and HCl are difficult to remove completely, and they result in acid mist formation.

The carbon yields were about 97% and a slight increase in density occurred. The conclusion is that the type of mobile unit being designed, when operated under the planned procedures, should give satisfactory results.

Materials of Construction

The principal item of concern in the regenerator design was the thermal insulation for the kiln. The system requires a high efficiency ceramic insulation which can withstand the anticipated vibrations of over-the-road travel as well as the heat and abrasion of the regeneration operations.

Extensive discussions with insulation manufacturers led to the conclusion that only silica-alumina fiber refractories could provide the combination of properties required. Samples of other materials and forms were tested. Alumina-silica coated with a hard surface proved to be the best but it was ultimately found that this refractory could not withstand the direct loading of the wet carbon. For this reason, the design was changed to provide a metal liner in the tube to protect the insulation.

The final step in this task evaluated material requirements for the liners in the kiln barrel and firing breech. The basic material selected was 300 series stainless steel. Since some material problems had been experienced in the hot corrosive sections of the laboratory unit, Inconel 601 was chosen for those areas in the mobile unit.

SECTION 4

PHASE II - COMPONENT SPECIFICATION DEVELOPMENT

With completion of Phase I, the basic system configuration had been delineated, the performance requirements of each system component had been defined, and weight and size limitations had been established. Taken collectively, these activities allowed the conclusion that a mobile carbon regeneration system could be assembled successfully, incorporating an onboard incinerator and scrubber to handle the materials stripped in the regeneration process.

The next step in the process was the preparation of detailed specifications for each component and subassembly. This effort was initiated with the understanding that commercially available equipment or modifications thereof would be specified wherever possible.

During the proposal stage and the early tasks of this program, the intent had been to modify an existing thermal treatment device for the basic regeneration unit. As the program progressed it became clear that this would not be possible. The requirements and limitations established for the rotary kiln, the incinerator and the scrubber were found to be outside the manufacturing limits of commercial suppliers. Thus, the first task in this phase was the development of a detailed design and manufacturing specifications for these three key items.

KILN

The kiln specifications had been set by the results of the laboratory-scale test program and the program goals. The principal factors were a 45.4 kg/hr throughput based on dry regenerated carbon weight and a final regeneration temperature of 982°C. With this starting point, a series of design calculations were made with the following results. For a 45.4 kg/hr recovery of dry regenerated carbon, the input will be 54.4 kg/hr of spent carbon together with 4.5 kg/hr of adsorbate and 36.3 kg/hr of water. Carbon losses will be 2.3 kg/hr due to burnoff and 6.8 kg/hr as unusable fines.

A temperature profile was constructed within the kiln barrel, starting with 100°C in the initial drying section and increasing to 982°C in the final regeneration stage. These temperature choices are based upon a 1093°C temperature for the tempered burner gas. Maximum temperature in the liner and firing breech was set at 1038°C with a 1260°C design temperature for the liner insulation. Maximum outside shell temperature was set at 93°C which requires a 731 Kcal/hr-m^2 heat loss.

Based upon these requirements and experience with the laboratory unit, materials of construction were selected. Carbon steel was chosen for the majority of components. For most of the elevated temperature components, low carbon 304 and 316 stainless steel were specified. Inconel 601 was chosen to provide greater high temperature strength for the hottest region of the kiln.

The kiln is a three stage rotary barrel with carbon flowing in one direction and hot gas in the opposite direction. Countercurrent gas flow provides for efficient heat utilization. In the first stage, wet spent carbon is cascaded by lifting flights into the hot gas stream. The contained water and low boiling adsorbates are removed here. In the second stage increasing temperatures are encountered which vaporize the remaining adsorbates begin thermal decomposition of some species. In the final stage, carbon is gently tumbled and the temperature is raised to the maximum of 982°C to complete regeneration.

To achieve a process flow of 45.4 kg/hr of dry carbon, a kiln barrel size of 371 mm inside diameter and 3.9 m long was calculated to be necessary. A slope of 0.6 cm/m and a rotational speed of 5 rpm at an 11.7% kiln loading gives the desired throughput. This is a 0.14 m^3/hr feed with a calculated 22 min residence time. The design operating parameters are tabulated in Table 11.

The major concern with the kiln was the thermal insulation. It must be lightweight, resilient, and have a high maximum temperature along with a high K factor. Originally it had been intended to use a ceramic kiln interior, but laboratory-scale testing made it clear that no available material could provide the necessary insulating properties and still withstand the abrasion and mechanical stresses which would be imposed. A metal inner barrel alleviated some of these problems.

The anticipated road stresses eliminated all of the normal rigid case or molded insulating media. Alumina-silica ceramic fiber forms were identified early in the program as the most promising material. Evaluation of commercially available materials resulted in choosing Kaowool for the kiln insulation. This was available as vacuum formed cylinders with a 7.25 kg/m^3 density. Based on its thermal ratings and the kiln characteristics, a minimum thickness of 114 mm would be required to maintain the 93°C design shell temperature.

The basic kiln must be fitted with several accessory items for operation. A feed unit is needed to deliver spent carbon to the first stage of the kiln. A 4 hr operating supply was chosen as the design point, which is equivalent to 0.57 m^3 of product. A feed system was chosen with an appropriately sized hopper and a dual pitch screw feed. A 58 mm pitch was chosen to meter the carbon from the hopper and a 76 mm pitch to provide movement through the transfer tube without plugging. An adjustable drive was added to allow delivery rates between 0.06 and 0.28 m^3/hr of wet dewatered carbon. The feed system used 300 series stainless for all components in contact with the wet carbon.

TABLE II. KILN DESIGN OPERATING PARAMETERS

Parameter	Identification	Value
Flowrates (kg/hr)	Carbon content of spent feed	54.4
	Adsorbates vaporized	4.5
	Residual water vaporized	36.3
	Carbon burnoff	2.3
	Carbon fines	6.8
	Carbon product recovered	45.4
	Fume gases to incinerator	272.2
Fuel (kg/hr)	Propane @ 1.3-2 psig	9.1
	Air @ 2-12% excess	150-168
Steam (kg/hr)		32-54
Pressure (mm)	Water Vacuum, @ Firing Breech	3.0-3.8
Temperatures (°C)	Ambient feed	21
	Initial drying section	100
	Final regeneration section	982
	Liners @ firing breech	1038
	Tempered burner gas	1093
	Liner and insulation rating	1260
	Fumes to incinerator	204
	Shell, @ 731 K cal/hr-m^2	93

At the other end of the kiln, a product recovery system is required to collect the regenerated carbon. This unit must cool the carbon, remove the unusable fines, and discharge the regenerated product.

Cooling is best accomplished by water quenching. Further, it can produce a slurry which is an effective form for both pumping and separating into size fractions. The selected design was a quench task using recirculated water. The slurry formed, normally about 7% solids, is pumped to a two deck screening separator. An air cooler was incorporated to insure adequate cooling before entering the separator. Carbon material larger than 40 mesh is removed in the first stage for recycle. Fines less than 325 mesh are separated in the second stage for disposal. The effluent is filtered to remove all particulates greater than 10 u and recirculated to the quench tank.

A slurry pump (Moyno) was selected to provide a flow of 0.6-0.9 m/sec. This was a compromise to provide high enough flow to prevent particle separation and plugging but below the rate which would cause exaggerated erosion of pipe and containment components.

The kiln is also fitted with a variable speed drive which permits adjustment of rotation between 1 and 10 rpm. This allows variations in the residence time which might be necessary for carbons with high loadings or difficult to remove adsorbates.

INCINERATOR/AFTERBURNER

Incineration of the offgases from the kiln had been selected as the most appropriate mechanism for detoxification and disposal of the desorbed contaminants. The gases would enter the incinerator from the kiln through a fume breech.

The initial design parameters were based upon a nominal fume inlet of 272.2 kg/hr effluent gases of which 4.5 kg/hr would be adsorbates or their decomposition products. Calculations gave 1.0 sec as an adequate residence time for mixing with burner gas and combustion.

For this residence, the firebox was sized at 0.61 m x 0.61 m x 1.5 m long. The associated burner was sized to provide up to 126,000 Kcal/hr using 11.3 kg/hr of propane and 227 kg/hr of air. These are maximum values, all components can be varied to achieve efficient combustion, based on stack gas composition.

The incinerator is designed so that fume gases enter below the burner to achieve good mixing. The design fume inlet temperature is 204°C with a flue gas outlet temperature of 982°C. The same temperature, 982°C, is the design temperature for the insulation hot face. The outer shell temperature maximum is 93°C.

The hot side of the incinerator is Inconel 601 to assure adequate corrosion resistance and hot strength. For insulation, alumina-silica Kaowool was chosen. A 3.63 kg/m^3 density wrap-around blanket form was deemed the

best choice with a mineral wool backup. After consideration of over-the-road stresses, an external layer of Kaowool board was added for mechanical strength. The overall insulation thickness was set at 142 mm in a carbon steel shell.

SCRUBBER

Although the incinerator is the primary means of degrading the offgases from the kiln, the products of combustion also may contain acid gases such as HCl and SO_2, particulate matter, and certain condensables. To remove the undesirable materials from the flue gases, a scrubbing system was felt to be necessary.

The scrubber consists of five components -- a spray tower, a caustic scrubber, a cooling tower, a liquor recirculator, and a stack blower.

Flue gases entering the scrubber system are adiabatically quenched in the spray tower, passed through the caustic scrubber to remove acid gases and into a packed tower to cool and demist. The gases venting from the packed tower should be composed primarily of CO_2, N_2, and O_2. Through monitoring of these gases, changes are made in the system, incinerator, and/or scrubber to correct any deficiencies. Vent gases are discharged through a stack using a blower to maintain an induced draft.

The recirculating scrubber liquor passes through a filter to remove particulate matter, through a cooler for temperature control, and into a makeup chamber to provide pH control. The scrubber design parameters are given in Table 12.

Material selection for the scrubber system was made principally for corrosion resistance except for the spray tower where elevated temperature strength was required. The material selected for the spray tower was Inconel 625. It was sized at 406 mm diameter and 1.8 m high to accomodate maximum inlet gas flow. It is flanged to the incinerator, thermally insulated and contains 6 spray nozzles spaced equally around the circumference.

The tower outlet discharges to a sump of approximately 380 L (100 gal) capacity. Anticipated operating conditions would have the sump two-thirds full of spent scrubbing liquor. The upper one-third acts as a duct to direct the quench gases into the caustic rubber.

The caustic scrubber is designed as a .61 mm diameter, 1.5-m-high cylinder packed within .15 mm of the top with Pall rings. The liquid distributor is located above the Pall rings. The scrubber is vented through a MSA mist eliminator using Teflon fiber spirals. The material of construction is fully annealed low carbon 316 stainless steel to minimize the possibility of chloride stress corrosion.

The liquor cooler is designed to handle 151,200 Kcal/hr (600,000 BTU/hr) using ambient air at 332 m^3/min (11,700 cfm). The cooler is located above the kiln at roof level to facilitate venting the cooling air.

TABLE 12. SCRUBBER DESIGN PARAMETERS

Parameters	Identification	Value
Gas Flows (m^3/min)	Spray column inlet	315
	Packed column inlet	15
	Scrubber effluent	8.4
Spray Water (L/min)	@ 50% vaporization	7.6
Scrubbing Liquor (L/min)	Scrubber inlet	114
	Bleed pH stream	7.7
	20% NaOH addition	5.7
Pressures (mm water vacuum)	Fan Suction	106.
	Scrubber outlet	51.
	Packed column inlet	25.
	Spray tower inlet	23.
Temperatures (°C)	Spray tower inlet gas	1016
	Packed column inlet gas	84
	Scrubber effluent gas	66
	Hot scrubbing liquor	79
	Cooled scrubbing liquor	57
	Ambient spray/caustic	21

The scrubbing system is designed to use available water with an onboard supply of caustic. Space and weight are available to carry 757 liters (a 7 day supply) of 20% NaOH solution. Although water would be drawn from a local source, the onboard system contains a receiver tank with a pressurized reservoir. This is to allow operation using adjacent water sources if a local pressurized supply is unavailable.

Water drawn from a local supply is discharged back to its source. Activated carbon filters are employed in line to insure acceptable quality for discharge.

REGENERATOR POWER AND CONTROLS

The total system was designed to draw power and gas from local utilities if available. Power requirements were estimated at 50 KVA. To compensate for peak needs, the design value was increased to 75 KVA using 240V 3-phase power.

The gas requirements for the kiln and incinerator combined were estimated at $.025 \times 10^6$ Kcal/hr. This is equivalent to 1.3 m^3/hr of propane.

It is expected that operation of the carbon regenerator will often be at a remote site where local utilities are unavailable. For this reason the system must be capable of independent operation. The gas requirements can be readily filled by liquefied propane trucked to the site. The 11.3 m^3/hr of gas is only 41.6 l/hr of liquid. A 1.25 to 2 psi pressure is adequate for the onboard system.

The electrical requirements can be met by an onboard diesel generator unit. The proposed physical arrangement allowed sufficient room in the forward part of the trailer for a commercial unit delivering 60 KVA continuously with a peak output to 75 KVA. The generator was located so that the exhaust could be ducted through the front of the trailer. The muffler was suspended on the front of the trailer above the cab of the tractor.

A diesel fuel storage tank was provided below the trailer deck. It was sized for 1514 liters (400 gal) to provide 3 days of continuous operation.

The diesel generator area was expected to generate sufficient noise and heat exclusive of the exhaust system that a soundproof enclosure would be required. Therefore, the generator enclosure was acoustically insulated. To maximize space utilization, the wall of the generator enclosure separating it from the main trailer area was used as the support for the control panel of the regenerator/incinerator.

The control panel contains the necessary instruments and recorders for operation of the regenerator. All components operated at elevated temperatures are continuously monitored by means of thermocouples. Gas flows, gas temperatures, and effluent gas compositions are also continuously monitored. Water temperatures, water flows, and pH are measured and

recorded. The diesel generator output is also instrumented. Fail safe interlocks are provided to protect against runaway reactions or other abnormal operating conditions. All interlocks are connected to visible and audible alarms. Where possible, interlocks are also connected to automatic shutoff-shutdown operators.

TRAILER DESIGN

In the early phases of the program a decision had been reached to base the trailer design on available configurations. Complete trailer design was felt to be beyond the scope of this program. The basis for the development was a standard enclosed platform van 2.44 m wide x 13.7 m long x 4.1 m high.

Four significant modifications to the standard design were found necessary:

(1) To reduce over-the-road stress on the regenerator/incinerator unit and allow at least limited off-the-road movement, the platform support was strengthened and reinforcements were added to minimize bed deflections.

(2) The sides and the rear of the van were redesigned so that they could be opened for ventilation when the trailer was positioned for operation. In the design chosen, the sides are made in two segments. The lower segment drops down to provide a walkway on both sides. The upper segment raises to provide an awning structure. Thus along with ventilation, the design improves access and movement within the trailer and worker protection during inclement weather.

(3) A separate access door is provided for the diesel generator enclosure. This area has its own overhead ventilation port.

(4) A series of jacks are incorporated to allow leveling of the trailer on sloped or uneven ground.

In addition to the above modifications, the trailer was fitted with several systems to accomodate specific components. Included in these are mounting supports for the generator muffler, the vent stack from the scrubber, and piping penetrations through the floor for water, gas and electricity. The underside of the trailer accomodates a series of tanks for water, diesel fuel, and caustic solution and the pumps associated with each of these services. The minimum road clearance required for these units was calculated to be 368 mm.

GENERAL REQUIREMENTS

The unit as designed is completely self-contained for field operation. It requires a parking area of 12.2 m by 24.4 m, a local source of fresh water, propane or natural gas, and a source of caustic replenishment. Power can be drawn from the local utilities or from its onboard generator. Also, a mechanism is necessary to dewater the spent carbon and drums are needed to receive the regenerated product.

SECTION 5

PHASE III - PROCUREMENT, ASSEMBLY, AND TESTING

With completion of the design phase, the acutal task of building the mobile regenerator was initiated. This involved the procurement of all of the required components, their assembly, the verification of system operation and finally delivery of the regenerator to the Environmental Protection Agency.

PROCUREMENT

The specifications prepared in the previous design phase were arranged in appropriate form for the preparation of purchasing documents. For all possibile components solicitation was for existing commercially available equipment. Where minor differences existed between requirements and available units, the differences were resolved in favor of the avilable equipment where possible. Where modifications were absolutely necessary, these were negotiated with suppliers. Although price was a consideration, performance was the major factor. Where modifications were necessary, the design requiring the least revision was the usual choice.

In all procurement and equipment selection the Quality Assurance Group at MSA was part of the decision process. They in turn verified that all incoming components complied with specifications.

In the course of the procurement process some problems with incoming equipment were revealed. In all cases the difficulties were minor and successfully resolved without compromising the system design or operational specifications.

These significant items are as follows:

(1) The carbon product separator as delivered exhibited unacceptable blinding (screen plugging). A vibrational antiblinding accessory was incorporated. In addition, to assist in effective separator operation, a 9.5 mm mesh basket screen was installed in the slurry tank.

(2) The spent carbon feed screw experienced some binding when the carbon contained an appreciable quantity of fines. Increasing the pitch of the feed tube essentially eliminated this difficulty. It was recognized, however, that carbon feed had to be reduced below the design level to prevent plugging when the feed contained a large percentage of carbon powder rather than granules. Two 6.4 mm water taps were added to facilitate washout operations in the feed mechanism.

(3) In the final portion of the kiln additional tumbling flights had to be added to get a gentler turning of the carbon and prevent fragmentation.

ASSEMBLY

With the delivery of the trailer, installation of system components was begun. With only minor changes, components were located as identified in the spatial arrangement. All equipment mounted within the trailer was bolted through the floor of the trailer to the steel cross members.

A common framework was installed below the trailer bed to support the 6 pumping units. Below deck supports were also installed for the 7 tanks; 3 for fuel, 2 for caustic and 2 for water.

Lugs were welded to the main trailer beams. The support work was bolted to the lugs. Pumps were bolted to the support work. Tanks were held in place by steel straps connected to the support work. Service piping was interwoven with the supports and passed through the floor to mate with the appropriate equipment.

As the assembly work progressed, certain accessories were added where a need was indicated. A slide control valve was added to the scrubber outlet to control the air from the blower. Roof hatches were incorporated over the two circulating cooler fans. Flexible mountings and connections were made at several points in the piping to minimize shock and twisting stresses.

ASSEMBLY REVIEW

Upon assembly of all components on the trailer, an operational plan was prepared and reviewed with the Technical Project officer at EPA. This included system drawings, flow sheets, operating parameters, and maintenance specifications. The system was approved with only a few minor changes in the arrangement and components. The Project Officer requested a level-measuring mechanism with each support jack and elapsed time meters for the kiln and the diesel generator for proper maintenance scheduling.

SYSTEM CHECKOUT

Following approval of the operating plan by EPA, a system shakedown was conducted using pure carbon containing only water. The system operated as designed with only minor changes required in the operating procedures. Operating conditions are given in Table 13.

TABLE 13. OPERATING CONDITIONS OF SYSTEM

Condition	Description
Regenerator Temperature	916°C
Incinerator Temperature	1010°C
Carbon Residence Time	45 minutes
Carbon Feed rate	.150 kg/hr (dry generated product)
Steam Input Rate	.156 kg/hr
Gas Input Rate	
CO_2 to regenerator	.142 kg/hr
N_2 to regenerator	.690 kg/hr
Air to incinerator	1.420 kg/hr
Water to scrubber	54.0 kg/hr

The only significant difficulty came from localized overheating in the kiln during heatup. The temperature excursions were not severe but they did trip automatic shutdown operators. This interlock was disconnected while leaving the alarms in place. Whether this interlock should be in place during system operation is a decision which will require some operating experience.

The successful shakedown run was followed by regeneration tests using contaminated carbon.

In preparation for the system demonstration, EPA had shipped to MSA drums of wet spent activated carbon used in the cleanup of a spill in Plains, VA. A preliminary run had been made with this carbon in the laboratory-scale regeneration unit during the design phase of the mobile regeneration unit.

The pollutant in the carbon was primarily Toxaphene ($C_{10}H_{10}Cl_8$) plus smaller quantities of other chlorinated compounds such as Aldrin ($C_{12}H_8Cl_6$), Dieldrin ($C_{12}H_9Cl_6O$), Heptachlor ($C_{10}H_5Cl_7$), and Chlordane ($C_{10}H_6Cl_8$). The carbon was discolored with a brown material that appeared as a thin layer on the outside surface of the granules. On the basis of bulk density measurements, the pollutant content was about 13% and water content was about 52% by weight. Both percentages are based on the dry weight of the regenerated product.

Two runs were made in which an 88% bulk volume yield of reactivated carbon was attained. The large loss is believed to be due to removal of the brown material from the surfaces of the granules. Table 14 presents the iodine numbers and bulk densities of the spent and regenerated carbons from these runs. The carbon was not identified, but on the basis of density and iodine number appears to be Pittsburgh type CAL carbon. If this is the case, the carbon was completely regenerated. All HCl was removed in the scrubber.

TABLE 14. IODINE NUMBERS

Carbon	Iodine No. (mg/g)		Bulk Density (g/cc)
	Run 1	Run 2	
Pittsburg CAL	960	960	0.500 dry
Spent Carbon	690	710	0.577 dry, 0.844 wet
Reg. Carbon, Run 1	1030	850	0.507 dry
Reg. Carbon, Run 2	960	940	0.510 dry

The success of these tests demonstrated the operating status of the mobile regeneration unit. On February 8, 1979 the unit was picked up by an EPA contracted hauler at the Evans City plant of MSA for transportation to the Hazardous Waste Engineering Research Laboratory at Edison, New Jersey.

Eight photographs are provided to give general views of the mobile regenerator/incinerator. Figure 4 shows the van closed for storage or transit. Figure 5 shows a rear view of the van open for operation. Carbon is being transferred from a drum into the carbon feed hopper. Figure 6 is a side view of the carbon feed and the kiln with the incinerator mounted above it. Figure 7 is a head end view of the kiln showing firing breech and gas burner. Figure 8 shows the side of the unit opposite the kiln with the scrubbing towers and water filtration units. Figure 9 shows the tank and pump assemblies slung beneath the bed of the trailer. Figure 10 is the control panel in the forward end of the van with the carbon screening unit in the right foreground. In Figure 11 the van is shown coupled to a stub tractor leaving the MSA plant enroute to EPA.

Figure 4. Enclosed trailer

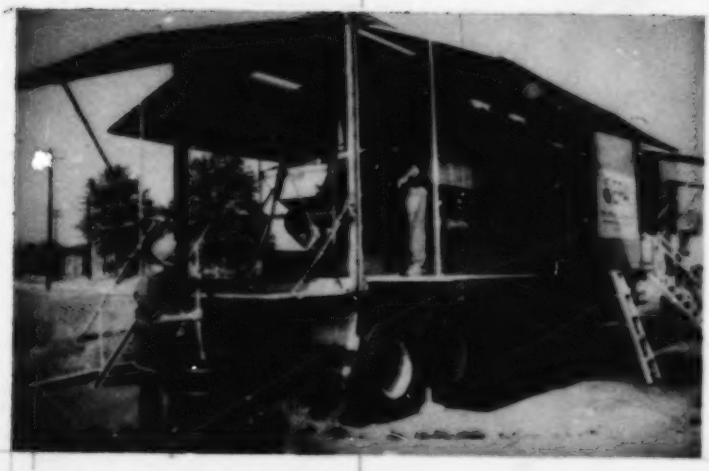

Figure 5. Trailer open for system operation

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Figure 6. Profile of kiln and incinerator

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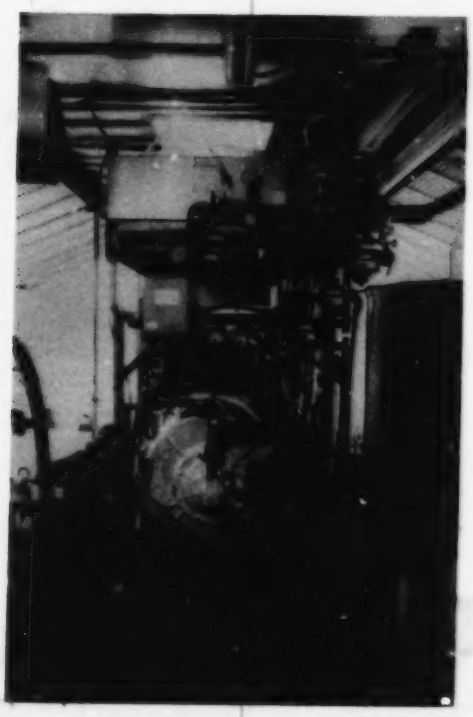

Figure 7. Head end of kiln

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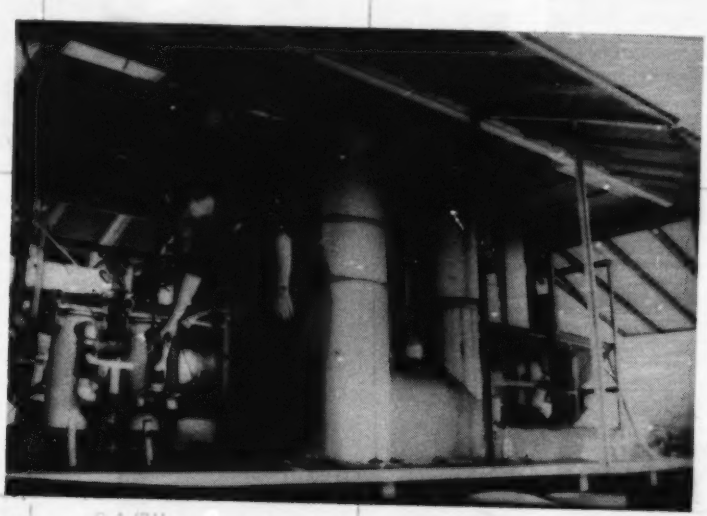

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Figure 8. Scrubber and filtration equipment

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Figure 9. Tanks and pumps beneath trailer bed

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Figure 10. Control panel and product screener

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Figure 11. Trailer in transit

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